CHARACTERISATION AND MODELLING OF STATIC RECOVERY PROCESS OF STAINLESS STEEL

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ABSTRACT

Annealing is a process where a material undergoes heat treatment for an extended time period and then slowly cooled. This process is very useful to alter the structure of the material where the grain size of the material tested change. The mechanical properties such as ductility and toughness also change during the annealing process. In this research, the recovery stage which is one of the annealing processes is being investigated. The material that has been use is type 304 stainless steel. At the recovery temperature which is in the range of $100^{o}C$-$400\ ^{o}C$, the behavior of stainless steel being investigated and the degree of softening, X_{rec} is calculated using Friedel's model. Friedel's model is a mathematical model used to calculate the degree of softening. There are 36 specimens being tested and from this experiment, 3 graphs plotted which are Xrec vs Time, X_{rec} vs Temperature and X_{rec} vs Pre-strain. Using Friedel's model, the activation energy, Q is calculated and being compared with other journal. The Q value obtained from X_{rec} vs Time and X_{rec} vs Temperature graphs are 466kJ/mol and 154kJ/mol respectively. There is no comparison made with X_{rec} vs Pre-strain graph. The Q value obtained from the graph plotted and from the journal is almost the same. So, the Friedel's model is valid to calculate the degree of softening at static recovery temperature of stainless steel. From this whole research, the behavior of stainless steel at static recovery temperature can be predicted using the model. This is very useful to apply at the real life problem such as buildings and structure that using stainless steel as material.

ABSTRAK

Sepuh lindap ialah proses dimana bahan akan melalui proses pemanasan pada jangka masa tertentu dan disejukkan secara perlahan. Proses ini sangat berguna untuk mengubahsuai struktur bahan dimana saiz bijian logam bahan yang diuji akan berubah. Sifat mekanikal seperti kemuluran dan kekuatan bahan juga berubah semasa proses sepuh lindap berlaku. Dalam kajian ini, peringkat pemulihan iaitu satu daripada proses sepuh lindap disiasat. Bahan yang digunakan ialah keluli tahan karat jenis 304. Pada suhu pemulihan iaitu dalam lingkungan 100^{o}C-400^{o}C, kelakuan keluli ini diperhatikan dan darjah kelembutan, X_{rec} dikira menggunakan model Friedel. Model Friedel ialah model matematik yang digunakan untuk mengira darjah kelembutan. Sebanyak 36 spesimen telah diuji dan daripada eksperimen ini, 3 graf telah diplot iaitu Xrec vs Masa, X_{rec} vs Suhu dan X_{rec} vs Tegasan. Dengan menggunakan model Friedel, nilai tenaga pengaktifan, Q boleh dikira dan dibandingkan dengan nilai yang terdapat pada journal. Nilai Q yang didapati daripada graf X_{rec} vs Masa dan X_{rec} vs Suhu ialah 466kJ/mol dan 154kJ/mol setiap satu. Tiada perbandingan dapat dibuat dengan graf X_{rec} vs Tegasan. Nilai Q yang didapati daripada graf yang telah diplot dan nilai yang terdapat pada journal adalah hampir sama. Oleh itu, model Friedel adalah sah untuk mengira darjah kelembutan pada suhu pemulihan keluli tahan karat. Melalui keseluruhan kajian ini, sifat keluli tahan karat pada suhu pemulihan boleh dijangka menggunakan model ini. Ini adalah sangat berguna untuk diaplikasikan dalam masalah kehidupan seharian seperti bangunan dan struktur yang menggunakan keluli tahan karat sebagai bahan.

TABLE OF CONTENTS

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

Annealing is one of the important processes in industry. Through this process, the mechanical properties of metals can be altered. This process is a heat treatment process where the material is changing in properties such as strength and hardness. It is a process that produces conditions by heating and maintaining a suitable temperature, and then cooling. Annealing is used to induce ductility, relieve internal stresses, refine the structure and improve cold working properties.

One of the processes in annealing is static recovery. The static recovery process occurs before the recrystallization temperature. At this stage, thermal energy is supplied to allow the dislocation to rearrange themselves into lower energy configuration. Through this experiment, the stainless steel will be going through several annealing process at the static recovery temperature and the results of this experiment will then use to develop a mathematical model.

Type 304 stainless steels are the most and widely used of many stainless steel. Although they have a wide range of corrosion resistance, they are not the most corrosion resistant of austenitic stainless steels. The chemical compositions of type 304 stainless steel are 0.08% C, 2% Mn, 1% Si, 18%-20% Cr and 8%-12% Ni. The 304 series of stainless steels exhibit high temperature strength, oxidation resistance, ease of fabrication and weldability, good ductility and good impact resistance down to at least -183^{o}C.

Table 1.1: Mechanical and physical properties of types 304 stainless steel [5].

Property	Type 304
Modulus of elasticity (GPa)	193
Tensile strength (MPa)	515
Yield strength (MPa)	205
Percent elongation at failure (%)	40
Melting temperature (^oC)	1400 -1450

1.2 PROBLEM STATEMENT

- To investigate static recovery effect in type 304 stainless steel when subjected to different strain.

1.3 PROJECT OBJECTIVE

- To validate Friedel's model of static recovery process of stainless steel in compression test.

1.4 PROJECT SCOPES

- Use types 304 stainless steels as test specimen.
- Operate lathe machine to shape the stainless steel into compression test specimen.
- Use box furnace to perform annealing.
- Perform compression test using compression test machine and gather required information about the test (pre-strain at 2.5%, 5%, 7.5%, and10%).
- Plot the graph using Microsoft Excel.

1.5 PROJECT BACKGROUND

Mechanical properties of stainless steel can be change by thermo-mechanical processes. This process required the stainless steel to go through some mechanical and annealing process. Annealing is one of the heat treatment processes that will be use in this research. Through this process, the stainless steel properties will be change such as ductility and hardness. The ductility of the material will increase while the hardness will decrease. The annealing process includes heating the material at suitable temperature and then cooling it slowly. There are three stages of annealing process which are recovery, recrystallization and grain growth. The static recovery process occurs at the temperature below the recrystallization temperature. At this stage, the thermal energy is supplied to allow the dislocation to rearrange themselves into lower energy configuration. The hardness of the stainless steel is reduced while the ductility increased. The stainless steel behavior at the recovery stage is then used to make a mathematical model. Through the compression test at different pre-strain (2.5%, 5%, 7.5% and 10%), a graph is then will be plot using Microsoft Excel.

CHAPTER 2

LITERATURE REVIEW

2.1 ANNEALING

The term annealing refers to a heat treatment in which a material is exposed to an elevated temperature for an extended time period and then slowly cooled. Ordinarily, annealing is carried out to (a) relieve stresses, (b) increase softness, ductility, and toughness and/or (c) produce specific microstructure. A variety of annealing heat treatment is possible; they are characterized by the changes that are induced, which many times are microstructural and are responsible for the alteration of the mechanical properties. [1]

Any annealing process consists of three stages (a) heating to the desired temperature, (b) holding or soaking at the temperature, and (c) cooling, usually to room temperature. Time is an important parameter in these procedures. During heating and cooling, there exist temperature gradients between the outside and interior portions of the piece; their magnitudes depend on the size and the geometry of the piece. [1]

Full annealing is relatively straightforward heat treatment in which the steel is heated to a temperature above the A_3 critical temperature and held at the temperature long enough to allow the solution of carbon and another alloying elements in the austenite. [3] The alloy is then furnace cooled; that is the heat treating furnace is turned off and both furnace and steel cool to room temperature at the same rate, which takes several hours. [1]

2.2 RECOVERY

The most subtle stage of annealing is recovery. No gross microstructural change occurs. However, atomic mobility is sufficient to diminish the concentration of point defects within grains and in some cases, to allow dislocation to move to lower energy positions. This process yields a modest decrease in hardness and can occur at temperatures just below those needed to produce significant microstructural change. [2]

The internal energy of the recovered metal is lower than that of the cold worked state since many dislocations are annihilated or moved into lower energy configurations by the recovery process. During recovery, the strength of a cold worked metal is reduced only slightly but ductility is usually significantly increased. [4]

2.3 RECRYSTALLIZATION

Upon heating a cold worked metal to a sufficiently high temperature, new strain-free grains are nucleated in the recovered metal structure and begin to grow, forming a recrystallized structure. After a long enough time at a temperature at which recrystallization takes place, the cold worked structure is completely replaced with a recrystallized grain structure. [4]

Primary recrystallization occurs by two principal mechanisms; (a) an isolated nucleus can expand with a deformed grain or (b) an original high grain boundary can migrate into a more highly deformed region of the metal. [4]

2.4 GRAIN GROWTH

After recrystallization is complete, the strain-free grains will continue to grow if the metal specimen is left at the elevated temperature; this phenomenon is called grain growth, Grain growth does not need to be produced by recovery and recrystallization; it may occur in all polycrystalline materials, metals and ceramics alike.

As the grains increase in size, the total boundary area decrease yielding an attendant reduction in the total energy; this is the driving force for grain growth. [1]

Grain growth occurs by the migration of grain boundaries. Obviously, not all grains can enlarge, but large one grows at the expense of small ones that shrink. Thus, the average grain size increases with time, and at any particular instant there will exist a range of grain sizes. [1]

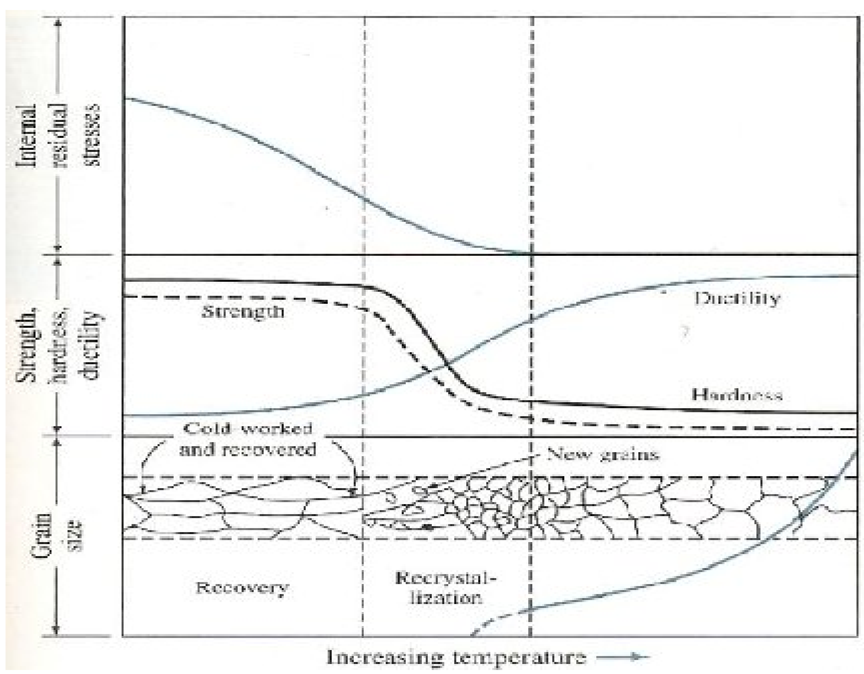

Figure 2.1: Effect of annealing on the structure and mechanical property changes of cold worked metal. [4]

2.5 COMPRESSION TEST

Compression test are determined by subjecting a specimen to an increasing compressive load until general yielding has occurred. Compression tests measure malleability (malleability is a measure of the extent to which material can withstand deformation in compression before failure occurs), and in compressive testing only compressive yield strength and compressive elastic modulus are measured. This is done in a manner similar method of tensile test. Theoretically, these values should be the same as tensile yield and modulus of elasticity values but, in reality, there is usually a small difference. [3]

In compression testing, the specimen barrels rather than necks. Because different factors are at work in barreling, malleable specimens flow in response to the load and actually compress rather than fracture. As the materials yield, it swells out (barrels), so that its increasing area continues to support the increasing load. [3]

Engineering stress, $\sigma = \dfrac{F}{A}$ [Eqn 2.1]

Where;

F= Instantaneous load applied perpendicularly to the cross section.

A= The original cross sectional area before any load is applied

Engineering strain, $\varepsilon = \dfrac{(l_i - l_o)}{l_o}$ [Eqn 2.2]

Where;

l_i = Original length before any load applied

l_o = The instantaneous length

Equation 1 and equation 2 are utilized to compute compressive stress and starain, respectively. By convention, a compressive force is taken to be negative, which yields a negative stress. Furthermore, since lo is greater than li , compressive strains computed from equation 2 are necessarily also negative. Compressive tests are used when material's behavior under large and permanent (i.e. plastic) strains is desired, as in manufacturing applications or when the material is brittle in tension.

2.6 STAINLESS STEEL

In all probability the most widely known and most commonly used material of construction for corrosion resistance is stainless steel. Stainless steels are iron based alloys containing 10.5% or more chromium. There are currently over 70 types of stainless steels. [5]

Stainless steel is not singular material, as its name might imply, but rather a broad group of alloys, each of which its own physical, mechanical, and corrosion-resistant properties. These steels are produced both as cast alloys [Alloy Casting Institute (ACI) types] and wrought forms [American Iron Steel Institute (AISI) types].

Generally, all are icon based with 12 to 30% chromium, 0 to 22% nickel, and minor amounts of carbon, columbium, copper, molybdenum, selenium, tantalum, and titanium. They are corrosion resistant and heat resistant, noncontaminating, and easily fabricated into complex shapes. [5]

2.6.1 Stainless Steel Classification

There are three general classification systems used to identify stainless steels. The first relates to metallurgical structure and places particular stainless steel into a family of stainless steels. The other two, namely, the AISI numbering system and the Unified Numbering System, which were developed by ASTM to apply to all commercial metals and alloys, define specific alloy compositions. The various stainless steel alloys can be divided into seven basic families: [7]

a) Ferritic
b) Martensitic
c) Austenitic
d) Precipitation Hardenable
e) Superferritic
f) Duplex (ferritic-austenitic)
g) Super austenitic

2.7 FRIEDEL'S MODEL

Friedel's model is a mathematical equation proposed by J.Friedel, Professor of Solid State Physics, University of Paris. This model is written in his book entitled Dislocations and was published by Pergamon Press in 1964 The Friedel's model is very useful in modeling the static recovery behavior of the materials. The mathematical equation is used to determine the degree of softening of the materials when some test is carried out. The degree of softening, X, from this model is being calculated from the expression; [6]

$$X_{rec} = \frac{(\sigma_m - \sigma_r)}{(\sigma_m - \sigma_o)} \qquad \text{[Eqn 2.3]}$$

Where;

σ_m = Flow stress immediately before unloading.

σ_r = Initial flow stresses recorded during reloading.

σ_o = Initial flow stresses recorded during pre-straining.

The relationship between the amount of recovery, time and temperature was found, over a wide range of conditions to be; [7]

$$X = c_1 \ln t - \frac{Q}{kT} \qquad \text{[Eqn 2.4]}$$

Where;

Q= Activation energy

k = Gas constant

c_1= Constant

T =Temperature

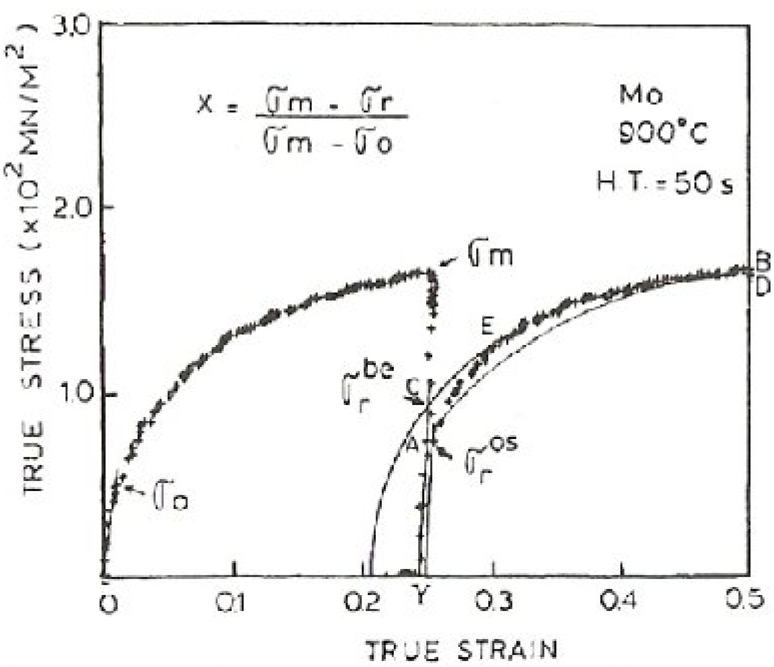

Figure 2.2: Determination of the reloading flow stress and the degree of softening by the back extrapolation and offset method. [10]

2.8 JOURNAL COMPARISON

To make sure this experiment works, a comparison with other journal being made. This is important to compare the calculation that being made with other established journal. Some of the journals are;

- H.L Andrade, M.G Akben, and J.J Jonas, Metallurgical Transaction. Effect of Molybdenum, Niobium, and Recrystallization and on Solute Strengthening in Microalloyed Steels **14(**1983),pp 1967-1977 [6]
- F.J Humphreys and M. Hatherly, Recrystallization and Related Annealed Phenomena Second Edition, UK, Elsevier Ltd, 2004 [7]
- T.Furu,R.Orsund and E.Nes,Subgrain Growth In Heavily Deformed Aluminium-Experimental Investigation and Modelling Treatment,Vol.**43**(1995),No.6,pp. 2209-2232 [8]

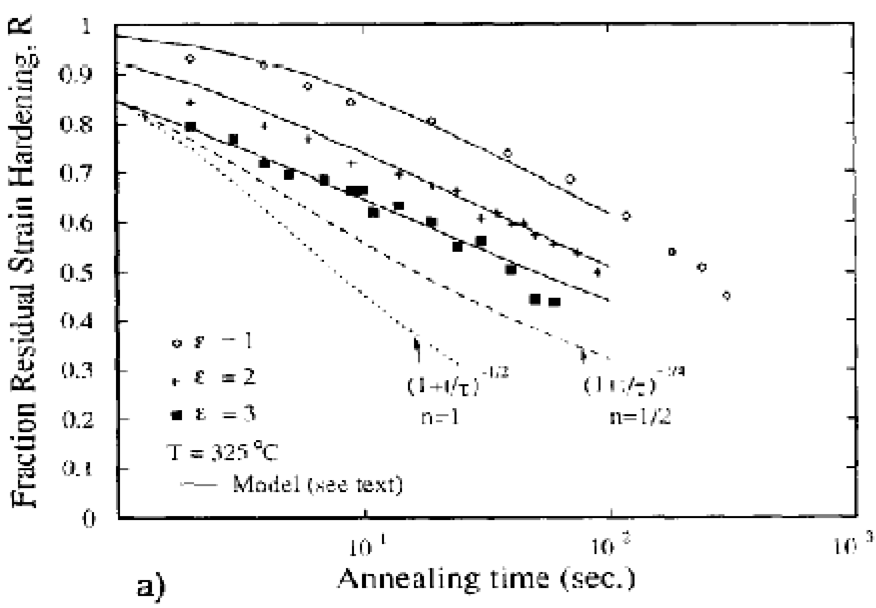

Figure 2.3: T.Furu, R. Orsund and E. Nes. Subgrain Growth in Heavily Deformed Aluminium-Experimental Investigation and Modelling Treatment, 1995 [8]

Table 2.1: Data collected from T.Furu, R. Orsund and E. Nes graph [11]

R (degree of softening)	Time(sec)
0.45	390
0.5	300
0.52	220
0.6	120
0.69	70
0.75	45
0.8	24
0.84	9
0.86	6
0.91	3.8
0.95	1.6

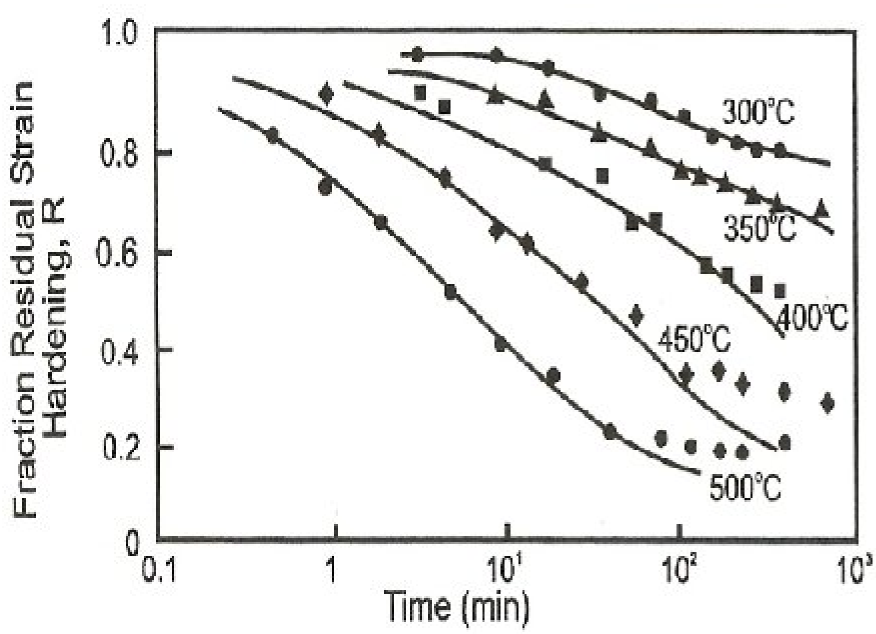

Figure 2.4: Modelling the recovery by Michalak and Paxton (Iron) [13].

Table 2.2: Data collected from J.T Michalak and H.W Paxton

R	Time(sec)
0.98	180
0.98	540
0.97	1200
0.9	2100
0.89	4200
0.87	6000
0.85	10800
0.83	13200
0.81	17400
0.81	27000

Table 2.3: Calculated values of Q from different journals

Material	Journal	Q (kJ/mol)
Stainless Steel	G.R. Stewart,J.J Jonas and F.Montheillet	405
Iron	J.T Michalak and H.W Paxton	105

CHAPTER 3

METHODOLOGY

3.1 FLOW OF METHODOLOGY PROCESS

Figure 3.1 shows the overall process of the experiment that being conducted. The function of this flow chart is to give guideline and information about the project. From this flow chart, the critical part of this research can be determined. The critical part of this research is the annealing process at the static recovery temperature. This part of this experiment must be conducted very well to get the accurate data. This is because, this research must be conducted at the temperature below the recrystallization temperature which is at the recovery state range between 100^{o}C and 400^{o}C.

The other critical part of this experiment is the design or dimension of the specimen for the compression test. The specimen's dimension for all 36 specimens must be the same. This is because the dimension is important during the compression test is being conducted. For this research, the dimension of the specimen must be 10mm in diameter and 25mm in length. The tolerance of the specimen is only $\pm 0.1mm$ If the dimension is not the same, the data obtained from the test is not accurate. So, the sequence of the flow chart must be followed to make sure this research is done and all the data from the test is accurate.

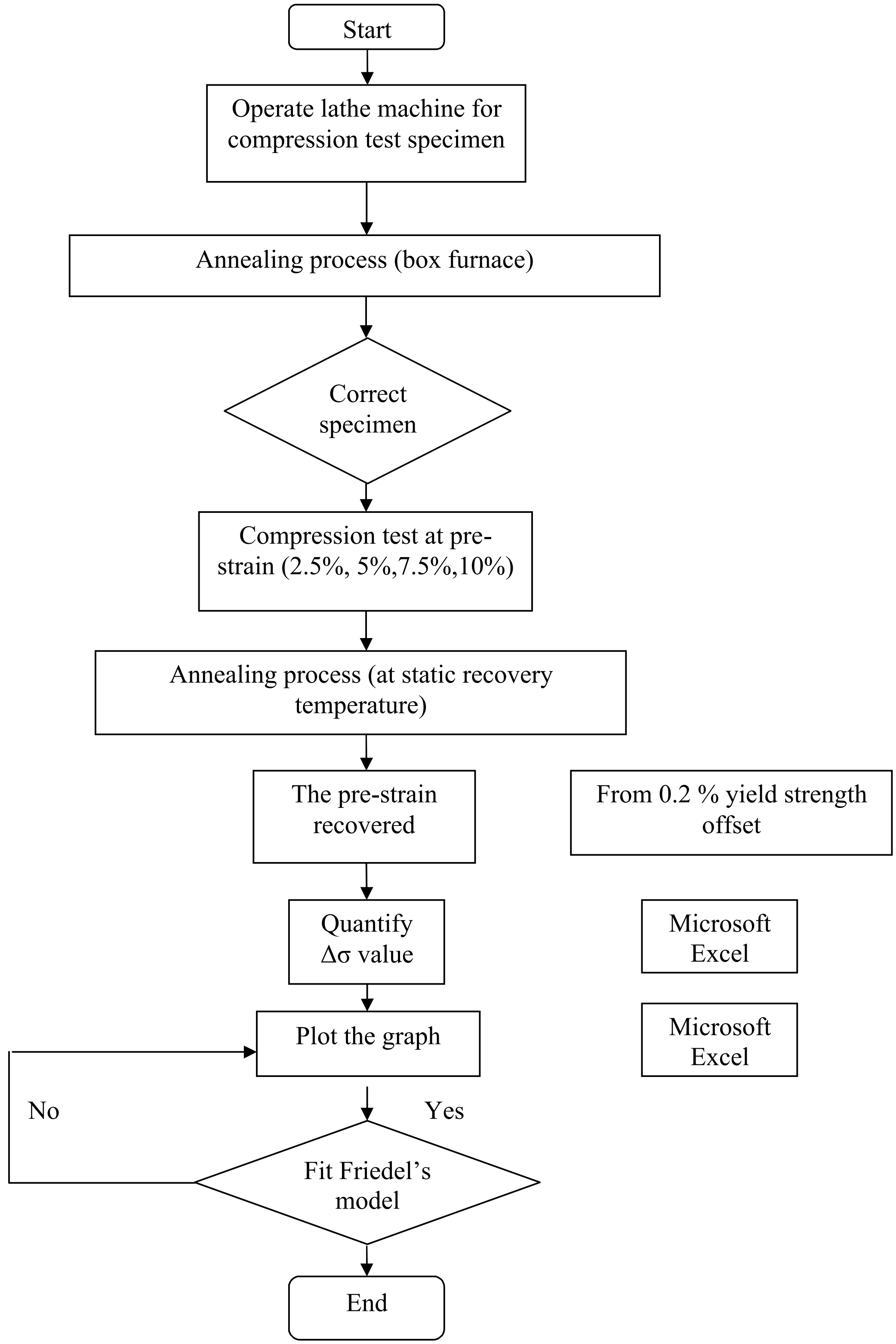

Figure 3.1: Flow chart of overall process

3.2 MACHINING PROCESS

Before the machining process can be start, the dimension of the specimen is being draw using Solid Work software. The entire specific dimension for the specimen must be known. The dimension of the specimen is 10mm in diameter and 25mm in length. The raw material used which is type 304 stainless steel must be cut with the band saw before using the lathe machine. This is because, the raw material comes from manufacturer in a long cylinder rod. So, to operate it using the lathe machine, the raw material must be cut first.

Figure 3.2: Bandsaw machine.

To use the band saw machine, cutting speed is a very important parameter. If not, the end product is not good. If the material used is hard, the cutting speed must be set lower. This is very important to avoid the saw from broke. The use of coolant is a must. This is because the coolant can protect the material and the saw from overheating.

Figure 3.3: Lathe machine

Figure 3.3 shows the conventional lathe machine used to prepare the specimens for the compression test. The important thing in handling the lathe machine is the speed needed to cut the specimen. If the speed of the lathe machine is correct, then the machining process can be carried out. If not, the specimen will damage and the finishing product is bad.

The dimension of the specimen must accurate which is 10mm in diameter and 25 mm in length. So, to produce a good specimen, the machine must feed slowly and the dimension of the specimen must be measure every time using the vernier caliper. Because of the length of the material which is stainless steel needed is only 25 mm, the machining process must be carried out very careful. Stainless steel is a hard material. So, it will take a long time to cut it little by little. The edge of the material which is very sharp must be taken out. The material will then undergo chamfering process to make sure it is safe for the user to use during the experiment.